UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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MEMORANDUM

TO: John Pavitt, Inspector
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FROM: Jed Januch, Environmental Protection Specialist
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Environmental Services Unit

QA REVIEWER: Susan Carrell, Washington State Department of Ecology __________

SUBJECT: Narrative for analysis samples collected during the Hanford Asbestos Sampling project

Project Code: ESD-250A
Account Code: 20122013B10P501E50

The following pertains to the quality assurance (QA) documentation associated with the asbestos analysis by stereomicroscope and polarized light microscopy (PLM) of twenty-two bulk samples of suspect asbestos building materials collected during the Hanford Asbestos Sampling project. I conducted the analysis using the U.S. EPA Region 10 standard operating procedure (SOP) for asbestos analysis ASB_001 and EPA method 600/R-93/116. For those tests for which the U.S. EPA Region 10 Laboratory has been accredited by the National Environmental Laboratory Accreditation Conference (NELAC), all requirements of the current NELAC Standard have been met.

The following comments refer to the quality control specifications for analysis of the following samples:

EPA Region 10 Sample Number	Description
12324001	272E/East of Shed - Transite fragment
12324002	272E/NE of Shed – Transite fragment
12324003	272E/along Atlanta Road – Transite fragment
12324004	272E/along Atlanta Road – Transite fragment
12324005	272E/along Atlanta Road – Transite fragment
12324006	2 West Steam Line Trailer – corrugated Transite fragment
12324007	2 West Steam Line Trailer – corrugated Transite fragment
12324008	2 West Steam Line Trailer – beige friable material
12324009	2 West Steam Line Trailer – beige friable material
12324010	Building 1717K – vinyl tile fragment
12324011	Building 183.6 KW – Transite fragment
12324012	Slight curve Transite
12324013	Building 183.6 KW – Transite fragment
12324014	Building 183.6 KW – Transite fragment

12324015	Building 183.6 KW – Transite fragment
12324016	Building 183.5 KE – Transite fragment
12324017	Building 183.5 KE – Transite fragment
12324018	Building 183.5 KE – Transite fragment
12324019	Building 1720 K – Transite fragment
12324020	Building 1720 K – Transite fragment
12324021	Building 1720 K – Transite fragment
12324022	Building 1720 K – Transite fragment

1.0 Holding time, Chain of Custody, and Sample Description

There is no recommended holding time for building materials analyzed for asbestos. The twenty-two bulk samples contained within plastic zip lock bags arrived at the laboratory in good condition with custody seals intact on August 10, 2012. I completed the analysis of the samples and associated QA samples on September 25, 2012. The EPA Region 10 Laboratory is a secure facility and the asbestos analysis area requires a key card for access.

I initially examined the samples visually and organized them into different building material types - Transite – cement asbestos board (CAB) and corrugated Transite (samples 12324001-12324007 and 12324011-12324022), vinyl tile (sample 12324010), and miscellaneous friable debris (samples 122324008 and 12324009). I have appended to this report several images of the samples as received by the laboratory. During the initial examination, I determined the Transite-CAB, corrugated Transite, and vinyl tile were generally non-friable however on closer examination, I noted that fragments of the corrugated Transite in particular appeared to have white colored fibrous material protruding from the edges and on weathered surfaces. The protruding fibrous material was friable in that I could easily dislodge it and separate it using finger pressure.

2.0 Results of Analysis

I detected chrysotile asbestos in all of the samples submitted for analysis except two (samples 12324008 and 12324009). Samples 12324008 and 12324009 contained fibrous glass in a carbonate matrix and organic material such as plant stems. Initially, I did not detect asbestos in the sample 12324010, but after matrix reduction for gravimetric analysis (gravimetry) I was able to detect a trace concentration (<1%) of chrysotile asbestos in this sample.

PLM examination confirmed the presence of chrysotile asbestos in twenty of the twenty-two samples submitted for analysis. I based my estimate of the concentration of asbestos for a subset of samples on gravimetry followed by 400-point counting. I estimated the concentration of asbestos for the remaining samples based on a calibrated visual estimate.

Samples 12324001 through 12324005 consisted of cement asbestos board (CAB) containing chrysotile asbestos. I performed gravimetry and point counting on samples 12324001 and 12324004 revealing a concentration range of 4.8% to 9.7% chrysotile asbestos. For samples 12324002, 12324003, and 12324005, I reported a visual estimate of 7% chrysotile asbestos.

Samples 12324006, 12324007, and samples 12324011 through 12324022 consisted of corrugated transite containing chrysotile asbestos. I performed gravimetry and point counting on samples 12324007, 12324011, 12324012, 12324018, and 12324022 revealing a concentration range of 6.6% to 8.9% chrysotile asbestos. For samples 12324006, 12324013, 12324014, 12324015, 12324016, 12324017, 12324019, 12324020, and 12324021, I reported a visual estimate of 8% chrysotile asbestos.

Table 1 displays a summary of the results of analysis for the 22 samples analyzed.

Table 1 - Summary of results of analysis for the Hanford Asbestos Samping Project, ESD-250A.

Sample No.	Sample Type	Preparation Method	Gravimetric Analysis	Asbestos Detected	Quantity (%)	Qualifier	Comments
12324001	Transite (CAB)	MP	X	chrysotile	9.7		Point Count
12324002	Transite (CAB)			chrysotile	7.0		Visual Estimate, SEM/EDS
12324003	Transite (CAB)			chrysotile	7.0		Visual Estimate
12324004	Transite (CAB)	FM	X	chrysotile	4.8		Point Count
12324005	Transite (CAB)			chrysotile	7.0		Visual Estimate
12324006	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324007	Corrugated Transite	FM	X	chrysotile	8.1		Point Count, SEM/EDS
12324008	Glass fiber in carbonate	MP	X	ND	ND	A	Visual Estimate
12324009	Glass fiber in carbonate	MP		ND	ND	A	Visual Estimate
12324010	Vinyl Floor Tile	FM	X	chrysotile	0.1	Trace	Point Count, SEM/EDS
12324011	Corrugated Transite	FM	X	chrysotile	6.6		Point Count
12324012	Corrugated Transite	FM	X	chrysotile	8.9		Point Count
12324013	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324014	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324015	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324016	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324017	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324018	Corrugated Transite	FM	X	chrysotile	6.9		Point Count
12324019	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324020	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324021	Corrugated Transite			chrysotile	8.0		Visual Estimate
12324022	Corrugated Transite	FM	X	chrysotile	7.5		Point Count

3.0 Sample Preparation

In most cases, preparation for analysis involves drying the samples in a drying oven. In this case, it was only necessary for the friable samples 12324008 and 12324009, which I dried in a muffle furnace for 4 hours at 60 degree centigrade (^{o}C). The remaining non-friable samples of cement materials (CAB and corrugated transite) and the vinyl floor tile did not require drying. It was necessary to break the non-friable samples into smaller pieces with the aid of tools including a metal hammer and a metal hoof nipper. I examined the freshly broken sections of each of the samples with the stereomicroscope. I collected images of the samples as I examined them under the stereomicroscope. I have appended the images to this report.

For qualitative analysis, I isolated suspected asbestos fiber bundles from each of the samples and mounted them in appropriate refractive index liquid for analysis by PLM for complete optical characterization. The optimum refractive index liquid for identification of chrysotile asbestos is high density with a refractive index (n_D) of 1.5500. I recorded the optical properties on electronic bench sheets.

For quantitative analysis, I selected eight samples representative of each type of material for further preparation for gravimetry and point counting. For the first sample of CAB, number 12324001, I ground a specimen with a corundum mortar and pestle using isopropanol as a grinding medium. This was time consuming and resulted in a fair degree of binder material remaining after acid treatment. I elected to mill the remaining nine CAB and corrugated transite samples in a SPEX model 6750 Freezer Mill. This method of grinding reduces the grain size and results in better acid dissolution to eliminate interference from binder material. The milling process involves cooling the samples in liquid nitrogen for 15 minutes and milling them for three x 2-minute cycles at a rate of 10 impacts per second resulting in a fine powder for analysis. I treated representative aliquots of each sample with a dilute hydrochloric acid solution for 10 minutes. I filtered the treated samples through a 0.4-micrometer (μm) polycarbonate (PC) filtering media with the aid of a glass vacuum filtration apparatus. I dried the treated residue and filter in a desiccant canister for 24-hours. Then I weighed the sample, subtracting the weight of the filter, and calculated the percent residue remaining after treatment.

Preparation of sample 12324010 (vinyl floor tile) required ashing in a muffle furnace for 6 hours at 450 ^{o} C. When cooled to room temperature, I used an analytical balance to weigh the sample to determine the amount of organic material eliminated. Then, I treated the sample with a dilute HCl solution in the same manner described above for the cement containing samples.

For point counting, eight slides were prepared for each sample by taking a small amount of residue from the surface of the PC filter and mounting it in refractive index liquid n_D =1.5500.

4.0 Asbestos Measurement System Calibration

I performed the calibration for the PLM and the refractive index liquids as required using appropriate methods and procedures. I checked the PLM daily to verify Köhler illumination and aligned the cross hair reticle using an anthophyllite reference slide. I verified that the values for the refractive index liquids used for this project were accurate on April 4, 2012, using an Abbe refractometer.

5.0 Analytical Procedures

I conducted analysis of samples for this project according to the U.S. EPA Region 10 SOP for asbestos analysis ASB_001 and EPA method 600/R-93/116. I performed PLM analysis using a Carl Zeiss Axioskop 40 PLM with a cross-hair reticle mounted in the microscope ocular. The magnification range for the Carl Zeiss Axioskop 40 PLM is 100 times (x) to 400x and 100x for dispersion staining.

Determination of asbestos involves evaluation of the morphology and optical properties of suspected asbestos fibers and fiber bundles. The raw data prepared for this project documents the gross sample description, stereomicroscopic observations, and optical properties observed by PLM including fiber morphology, extinction angle, sign of elongation, birefringence, and central-stop dispersion staining characteristics in appropriate refractive index liquids.

6.0 Quality Assurance and Quality Control

I reviewed a set of commercially prepared slides as standardized references. I also analyzed a specimen of chrysotile asbestos standard reference material (SRM) 1866b obtained from the National Institute of standard and Technology (NIST). During PLM analysis, method blanks were prepared daily to determine that refractive index liquid and tools used for this project were asbestos-free. All supplies and tools were determined to be asbestos-free.

The EPA Region 10 Laboratory routinely participates in performance testing sample analysis as well as routine round robin inter-laboratory sample exchanges. In addition, a certain percentage of samples are selected for intra and inter-laboratory analysis. For this project, the asbestos analysts at the National Enforcement Investigations Center (NEIC) in Denver, Colorado performed inter-laboratory duplicate analysis on three samples. Table 2 is a summary of the results of the quantitative QA analysis for this project.

Table 2 - QA Samples

Intra Laboratory Duplicates				QA sample Type				
12324004	Transite (CAB)	FM	X	Intralab duplicate	chrysotile	4.9		Point Count
12324008	Glass fiber in carbonate	MP	X	Intralab duplicate	ND	ND	A	Visual Estimate
12324022	Corrugated Transite	FM	X	Intralab duplicate	chrysotile	7.5		Point Count, SEM/EDS
Inter Laboratory Duplicates								
12324007	Corrugated Transite	FM	X	Interlab duplicate	chrysotile	33.0		Visual Estimate
12324012	Corrugated Transite	FM	X	Interlab duplicate	chrysotile	12.0		Visual Estimate
12324018	Corrugated Transite	FM	X	Interlab duplicate	chrysotile	18.0		Visual Estimate
Region 10 Supplemental Duplicates								
12324007A	Corrugated Transite	MP	X	Intralab duplicate	chrysotile	13.3		Point Count
12324007B	Corrugated Transite	MP	X	Intralab duplicate	chrysotile	15.0		Point Count
12324007C	Corrugated Transite	MP	X	Intralab duplicate	chrysotile	14.4		Point Count
12324007D	Corrugated Transite	MP	X	Interlab duplicate	chrysotile	17.1		NEIC REDO, Point Count
NEIC Supplemental Duplicates								
12324007REDO	Corrugated Transite	FM	X	Interlab duplicate	chrysotile	7.2		R10 REDO, Point Count
12324007P1	Corrugated Transite	P	X	Interlab duplicate	chrysotile	25.1		Point Count
12324007P2	Corrugated Transite	P	X	Interlab duplicate	chrysotile	23.4		Point Count
12324007P3	Corrugated Transite	P	X	Interlab duplicate	chrysotile	21.2		Point Count

In addition to PLM analysis, I used alternate methods to verify morphology and particle chemistry by scanning electron microscope (SEM) with energy dispersive spectroscopy (EDS) and verify mineral type and semi-quantitative analysis by x-ray diffraction (XRD).

Intra Laboratory Precision

Prior to analysis, I selected three samples (12324004, 12324008 and 12324022) for intra (within) laboratory duplicate analysis. All three samples were prepared for analysis in the same manner. I detected chrysotile asbestos in samples 12324004 and 12324022, but not in sample 12324008. The relative percent difference (RPD) between the result of analysis for sample 12324004 and its duplicate were within 2.06% and for sample 12324022 the RPD between the parent sample and the duplicate was 0%. I did not detect asbestos in either sample 12324008 or the duplicate.

Inter Laboratory Precision

I sent three samples (12324007, 12324012, and 12324018) to NEIC for inter-laboratory duplicate analysis. While both labs detected chrysotile asbestos in all three of the duplicate samples, the quantity of asbestos reported differed (RPD 29.7% - 121.17%). The variation in results was apparently due to preparation and analytical techniques used by the laboratories.

A certain degree of variation between laboratories is common for asbestos quantitation due to heterogeneity of samples, especially in instances where different sample preparation and analytical techniques are used for nonfriable materials. In this case, the QA officials at the EPA Region 10 Laboratory and the NEIC Laboratory agreed that one sample (number 12324007) should be reanalyzed in triplicate using preparation techniques other than freezer milling. We are speculating that the freezer mill, while useful for reducing grain size for more efficient acid dissolution, may result in reducing the size of asbestos fibers to the extent that they either may be too small to be resolved by PLM or possibly rendered so small that they pass through the 0.4-micron filter during acid dissolution. For reanalysis, the EPA Region 10 Laboratory replicate samples were lightly ground in a corundum mortar and pestle with isopropanol grinding medium followed by treatment with dilute HCl solution. The NEIC Laboratory replicate samples were crushed with a pliers followed by treatment with dilute HCl solution. In both cases, the final qualitative result identifying chrysotile as the asbestos mineral present in the samples remained the same. The quantitative results on the original QA analysis as well as the QA reanalysis show that the concentration of chrysotile asbestos was greater than 1% and the average result for reanalysis of sample 12324007 for each laboratory appeared closer (RPD=48%).

Analysis by Alternate Methods

Analysis by SEM/EDS verified the morphology of the chrysotile to be asbestiform and the chemistry (major concentration of Si and Mg) to be consistent with chrysotile. I collected images of chrysotile asbestos by SEM that I have appended to this report along with EDS spectra collected during examination of the chrysotile structures.

Analysis of sample 12324007 by XRD confirmed the presence of clinochrysotile (chrysotile) as well as calcite, aragonite, magnetite, quartz, and amorphous material. I estimated the concentration of chrysotile in sample 12324007 at 23.73% based on peak area and reference intensity ratios. I will provide a narrative report describing the results of analysis by XRD for sample 12324007 in a separate data package.

7.0 Reporting Limits / Data Qualifiers

The detection limit for asbestos minerals by PLM using EPA method 600/R-93/116 is approximately 1%. I am reporting the sample results for this project as the average percentage based on gravimetry and point counting by PLM or based on a calibrated visual estimate by comparison with similar samples analyzed by gravimetry and point counting by PLM. If the component is present but no percentage is reported, the qualifier for present but not quantified (PNQ) is used. If the component is not present, the qualifier for absent (A) is used. If the component has been positively identified, but is estimated to be less than 1%, the qualifier used is trace (T).